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INFLUENCE OF ACTIVE GAS CONTENT IN POWDER ON MECHANICAL PROPERTIES OF WORKPIECE BLANKS PRODUCED BY PLASMA ADDITIVE TECHNOLOGIES

Purpose. *Analysis of the influence of powder quality (content of active gases) on the level of mechanical properties of workpieces produced by plasma additive technologies.*

Research methods. *Chemical composion of the powders was determined in accordance with regulatory document.*

The presence of non-metallic inclusions and contaminants in the powders was determined visualy.

To quantify the oxygen in the powders, the vacuum extraction method was used in accordance with ДСТУ ISO 4491-4:2016. Oxygen and nitrogen gas analyser TC 500 (company LECO). Before being loaded into the dosing unit, all powders were dried in an oven (temperature - 250°C, for 1 hour).

Samples (size 130×70×14 mm) were produced by additive growth by plasma powder surfacing *(PPS) on a specialised robotic device STARWELD 190H.*

The chemical composition of the grown samples was determined by the spectral method using a SPECTROMAX optical emission spectrometer according to standard methods in accordance with ГОСТ19863.1-19863.12.

The content of nitrogen and oxygen in the deposited metal was determined in accordance with TC 500 (company LECO).

Microstructural analysis were carried out on the samples in the longitudinal and cross directions before and after heat treatment, after etching in a reagent for electrowinning of heat-resistant alloys (H_3PO_4 – 800 ml +CrO3 – 100ml).

The mechanical properties of the alloy were obtained on standard cylindrical samples according to ГОСТ 1497-84, ГОСТ 1497-84, ГОСТ 10145-81s.

Results. *The chemical composition of the deposited metal is determined by the chemical composition of the powder. The content of oxygen and nitrogen in the deposited metal is determined by the content of these elements in the powder itself, as well as by the protection of the weld pool with argon. It has been established that in the alloy ЭП-648ВИ when the powder contains nitrogen in the amount of [N] ≥ 0.03 wt. %, or oxygen in [O] ≥ 0.02 wt. % in the deposited metal at a temperature of 1100 °C, a significant decrease in its deformation capacity (ε ≤ 3.8 %) is recorded, which under the influence of thermal stresses leads to the appearance of hot cracks, and as a result, we see a significant decrease in the strength and plasticity in the longitudinal direction.*

Scientific novelty. *The influence of the quality of the alloy ЕП-648ВИ powder on the properties of workpieces obtained using additive technologies has been established.*

Practical value. *The negative effect of the content of active gases on the mechanical properties of the grown workpieces, established in this work, will allow to screen out powder with an unacceptable content of active gases ([N] ≥ 0.03 wt. %, or oxygen in [O] ≥ 0.02 wt. %) at the stage of incoming inspection.*

Key words: *additive technologies, plasma surfacing, mechanical propities, aircraft engines, growing.*

Introduction

Additive growth processes by microplasma deposition using powder can be thought of as melting processes with a small bath and short crystallisation times. The melt bath dimensions are in the range of 10-1000 μm, and the cooling rate can reach 10^6 K/s. Thus, the theory of welding and rapid curing can be used to

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describe the process and curing conditions in additive manufacturing [1].

Like most complex alloys, nickel-based alloys solidify within a temperature range (soft zone) determined by the liquidus temperature (beginning) and the solidus temperature (end of solidification). This is due to the different solubility of elements in liquid and solid state, which leads to an increase in emissions from the initial liquid composition and a decrease in the liquidus temperature during solidification [2].

As long as the diffusion rate is sufficiently high to maintain thermodynamic equilibrium at the solidification front with the actual liquid temperature, the flat morphology of the solidification front is maintained and growth occurs [3]. Cooling rates in casting processes and powder bed additive growth processes are typically too high [4]. The actual temperature at the solidification front falls below the local liquidus temperature and thermodynamic equilibrium cannot be maintained. The flat solidification front becomes unstable and cells begin to form. With a further increase in the degree of constitutional hypothermia, dendritic morphology is favoured over cellular morphology. As long as the heat is dissipated through the solid, directional growth takes place and a columnar dendritic solidification structure develops. Once the constitutive supercooling becomes high enough so that heat is dissipated through the liquid, multiple nucleation centres can form and equirectly axial dendritic growth begins [2, 5]. As the curing rate increases, the potential for maintaining thermodynamic equilibrium through diffusion decreases, and the range of natural supercooling increases. This is reflected in the influence of two combined parameters on the curing structure: the ratio of the temperature gradient and the curing rate

The specifics of transfer, heating and melting of the filler material (powder) primarily distinguish microplasma powder surfacing from other surfacing methods with a consumable and non-consumable electrode [6].

The thermal cycle can be divided into a crystallisation process with liquid-solid interaction and a process that takes place in the solid state after complete crystallisation [7].

The solidification process is determined by the solidification conditions at and around the solidification front as well as the elements involved and the forming phases. The solidification conditions are a consequence of the melt bath geometry and the process conditions (liquid volume, heat flow and direction, etc.). In combination with the thermophysical properties of the elements present (liquid and solid solubility, diffusion coefficients, etc.), both aspects determine the morphology of the solidification front. From there, the type and scale of the curing structure (microstructure) [2, 8].

The process of growing workpieces by microplasma powder surfacing (MPS) can be represented as a set of the following technological stages of a nickel heat-resistant alloy that have a significant impact on the properties of the grown workpiece:

1) production of cast metal by vacuum induction smelting;

2) producing filler powder by spraying a cast billet;

3) applying a certain amount of deposited metal to the surface of the workpiece to be grown by means of compressed arc surfacing [9].

The first two technological stages affect the chemical composition of the deposited metal and impart a certain ‘heredity’ in the form of the content of impurity elements of oxygen and nitrogen. This will ultimately have a significant impact on the welding and technological properties of the dispersed additive, the energy conditions for the formation of the weld pool and the specified cross-section of the metal to be deposited. The third process step has an impact on the stress-strain state and technological strength of the grown workpiece [10].

Problem statement

The use of plasma powder surfacing is a promising method of manufacturing workpieces for aircraft engines, which allows for high accuracy of geometric shapes and reduced material waste. However, the process of forming the deposited metal is accompanied by a number of challenges that affect the quality of the final parts. One of the main factors affecting the mechanical properties of products is the chemical composition of the powder, in particular, the content of active gases such as oxygen and nitrogen.

The urgency of the task is the need for an in-depth analysis of the influence of the chemical composition of powders on the processes of microplasma surfacing of heat-resistant nickel alloys. It is known that an excessive amount of oxygen and nitrogen in the powder leads to a decrease in plasticity, increased brittleness and the occurrence of cracks in the deposited metal. This, in turn, significantly limits the use of such materials under high mechanical and thermal loads.

The aim of the study is to determine the relationship between the content of active gases in powders and the structure and properties of the deposited metal, as well as to assess their influence on the level of mechanical characteristics of the workpieces obtained by plasma additive growth.

Research objectives:

- analysis of the chemical composition of powders and deposited metal to determine the content of active gases;
- assessment of the influence of oxygen and nitrogen on the crystallisation processes of the deposited metal, in particular on the formation of the microstructure and its defects;
- investigation of the relationship between the content of active gases in powders and the mechanical properties of workpieces grown by microplasma surfacing;
- development of recommendations for ensuring the

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stability of mechanical properties by controlling the content of active gases in powders.

The results of the study are expected to identify the permissible limits for the content of active gases in powders and develop recommendations for improving the plasma powder surfacing technology. This will help to improve the reliability and performance of parts produced by the additive growth method, in particular for use in aircraft engines.

Results and discussion

The objects of study were powders from the nickel-based heat-resistant alloy ХН50ВМТЮБ-ВИ (ЭП648ВИ) of five different batches. The chemical composition and content of oxygen and nitrogen in the powder are given in tables 1 and 2.

As can be seen from Table 2, the oxygen content is highest in powder № 5 (O_2=0.026%), and lowest in powder №2 (O_2=0.0054%). The nitrogen content is highest in powder № 1 (N_2=0.0358 %), and the lowest in powder № 3 (N_2=0.0191%).

Table 1 – Chemical composition of powders

№	Content of elements, %						
	Mo	W	Cr	Al	Nb	Ti	Ni
1	3.25	4.9	33.7	0.6	1.0	0.7	base
2	2.9	4.7	32.8	1.0	1.0	0.96	
3	3.3	5.0	33.2	1.0	1.1	1.0	
4	3.0	4.9	34	0.7	0.9	0.8	
5	3.2	5.0	33.8	0.9	0.8	0.9	

Table 2 – Gas content of powders

№	Gas content, %	
	O_2	N_2
1	0.0196	**0.0358**
2	0.0054	0.0271
3	0.0085	0.0191
4	0.015	0.0215
5	**0.026**	0.0230

In addition, it should be noted that the high content of oxygen and nitrogen in powders can be the result of insufficiently controlled conditions of their manufacture or storage. It is important to bear in mind that even minimal fluctuations in the content of these gases can have a significant impact on subsequent stages of the technological process, including the formation of the deposited metal and its mechanical properties.

The chemical composition of the deposited metal and gas content are presented in tables 3, 4.

Table 3 – Chemical composition of the deposited metal

№	Content of elements, %						
	Mo	W	Cr	Al	Nb	Ti	Ni
1	3.12	4.72	34.6	0.62	0.98	0.72	base
2	2.97	4.81	33.6	0.97	1.03	0.98	
3	3.23	4.87	34.2	0.9	1.07	0.96	
4	3.08	4.97	33.4	0.72	0.93	0.83	
5	3.13	4.9	33.2	0.93	0.81	0.88	

From all five powders, samples of the following sizes were grown on the STARWELD 190H using the MPS method at the same modes 140/70/14 mm.

Table 4 – Gas content in the weld metal

№	Gas content, %	
	O_2	N_2
1	0.02436	**0.0818**
2	0.0068	0.03323
3	0.1093	0.02267
4	0.0187	0.02715
5	**0.031**	0.0290

After growing, the samples were heat treatment in the following modes: hardening (T_h = 1140 ± 10°C, τ = 4 hours, air cooling); ageing (T_a. = 900°C, τ = 16 hours, air cooling).

From tables 1 and 3, we can see that the difference between the chemical elements in the powder and in the deposited metal is insignificant and is within the range of 1-5%. Therefore, it can be concluded that the chemical composition of the deposited metal is determined by the chemical composition of the powder.

It should be noted that even minor deviations in the chemical composition, such as increased oxygen or nitrogen content, can affect the final properties of the product, contributing to the formation of microstructural defects, such as carbides or nitrides. This emphasises the need to optimise the composition of powders to reduce the impact of impurities.

From tables 2 and 4 and the shielding gas, we can see that the difference between the oxygen and nitrogen content of the powder and the deposited metal is in the range of 20-30%. Based on this, it can be concluded that the content of oxygen and nitrogen in the deposited metal is determined by the content of these elements in the powder itself, as well as by the protection of the weld pool with argon (quality of argon and stability of its supply).

It should be noted that an increase in the content of active gases in the deposited metal compared to the powder can also be caused by technological factors of external contamination or insufficient equipment tightness. This feature requires not only enhanced control over the shielding gas supply conditions, but also proper purity of the argon used during surfacing.

The samples were made after heat treatment, the tests were carried out in the longitudinal and cross directions relative to the growth layers, and the following results were obtained [11, 12]:

for powder №1:

- cross direction: σ_u= 878.1 MPa; $\sigma_{0.2}$= 532,1 MPa; δ= 16.4 %; ψ= 15.2 %;
- longitudinal direction: σ_u = 443.9 MPa; $\sigma_{0.2}$= 485.1 MPa; δ=2.2 %; ψ= 5.9 %;

for powder №2:

- cross direction: σ_u = 918.3 MPa; $\sigma_{0.2}$ = 625.2 MPa; δ= 20.6 %; ψ= 26.0 %;

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- longitudinal direction: σ_u = 873.1 MPa; $\sigma_{0.2}$= 457.1 MPa; δ=13.8 %; ψ= 15.8 %;

for powder №3:

- cross direction: σ_u = 883.0 MPa; $\sigma_{0.2}$= 633.1 MPa; δ= 23.7 %; ψ= 42.3 %;
- longitudinal direction: σ_u = 906.5 MPa; $\sigma_{0.2}$= 689.9 MPa; δ=12.8 %; ψ= 17.8 %;

for powder №4

- cross direction: σ_u = 945.7 MPa; $\sigma_{0.2}$= 645.8 MPa; δ= 18.7 %; ψ= 24.7 %;
- longitudinal direction: σ_u = 799.7 MPa; $\sigma_{0.2}$= 535.1 MPa; δ=9.2 %; ψ= 16.8 %;

for powder №5:

- cross direction: σ_u = 883.0 MPa; $\sigma_{0.2}$= 633.08 MPa; δ= 23.7 %; ψ= 42.3 %;
- longitudinal direction: σ_u = 575.3 MPa; $\sigma_{0.2}$= 501.8 MPa; δ=2.8 %; ψ= 9.1 %.

It should be noted that for specimens № 1 and № 5, we can see a significant decrease in strength in the longitudinal direction (for № 1 – more than two times; for № 5 – one and a half times), and plasticity is practically absent. The cause of the brittle fracture of the longitudinal specimens are the cracks detected during the metallographic examination. During testing, the load on the longitudinal specimens is applied perpendicular to the cracks, which leads to their development and subsequent fracture of the specimens. Metallographic studies confirm that the high content of active gases in the powder contributes to the formation of cracks in the deposited metal, which are concentrated at the grain boundaries. This directly affects the ductility and strength in the longitudinal direction. On the contrary, powders with a minimum content of active gases demonstrate better mechanical properties due to a reduction in the likelihood of defect formation.

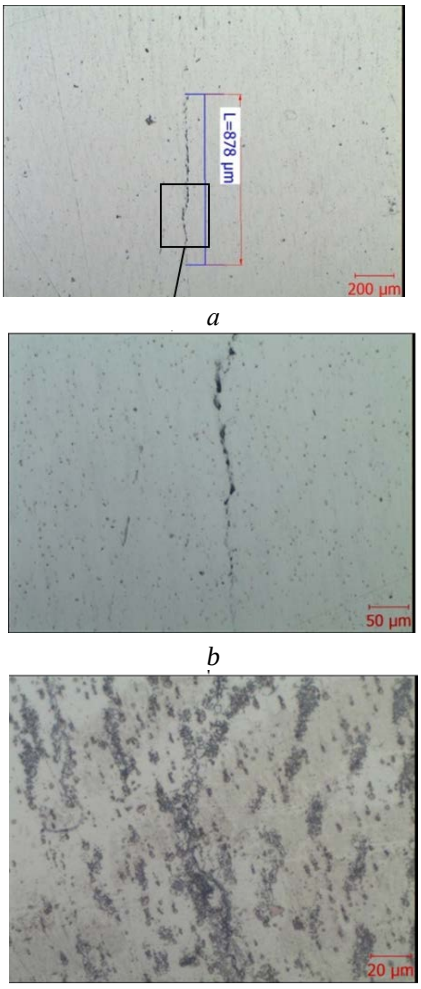
On cross-sectional specimens, the stress is applied along the cracks, so they do not have a significant impact on the mechanical properties.

Metallographic examination of the microgrooves of samples № 1 and № 5, made in longitudinal and cross sections, revealed cracks.

After electrolytic etching of the grinds in a 10% oxalic acid solution, it was found that the cracks developed along the grain boundaries along the heat sink, across the layers (fig. 1). In the areas of crack propagation, accumulations of large carbides along the grain boundaries are observed (fig. 2). It should also be noted that the crack surfaces have smoothed melted edges.

The fracture fractogram shows that almost the entire fracture surface (~98 %) of the opened crack is characterised by a smoothed «melted» surface, which is typical for hot crack fractures (see fig. 2). There are small individual areas with a cellular structure, which is characteristic of plastic fracture (see fig. 2).

The fractographic analysis also shows the importance of proper thermal cycle control during the surfacing process. The cyclical heating typical of repeated surfacing can increase the tendency to crack formation, especially in materials with high oxygen and nitrogen content. Optimising cooling and heat treatment conditions can significantly reduce the risk of such defects.



a

b

c

Figure 1. Crack on the samples: *a* – unetched grinding, ×50; *b* – unetched grinding, ×200; *c* – grinding after etching, ×500

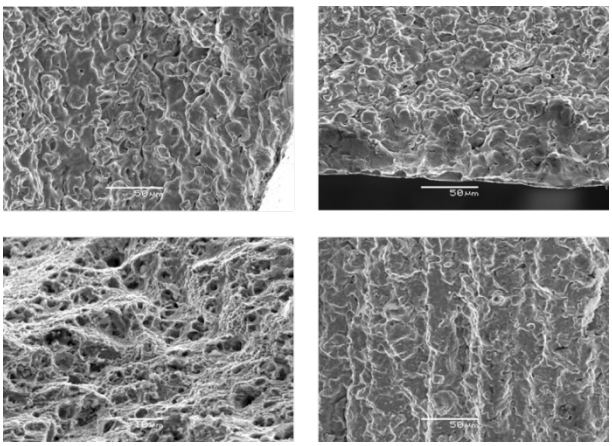


Figure 2. Fracture pattern of an open crack

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It is known that in the process of multilayer surfacing, the surface of several previous rolls under the weld pool is reheated to temperatures of 1100-1200°C (fig. 3), and under conditions of transverse vibrations, such cyclic reheating can also be present for the already pre-crystallised metal behind the weld pool of the deposited roller [13, 14]. Under these conditions, the tensile welding deformations with residual stresses are added up in the above-mentioned zones of the deposited metal. In the case of a sufficiently high stiffness of the additive structure, when their total value exceeds the relatively small deformation capacity of the deposited metal with a high content of dissolved gases within the temperature of 1100°C, during repeated heating of the deposited metal, its destruction occurs by the mechanism of «ductility dip cracking» (classification according to [15-16].

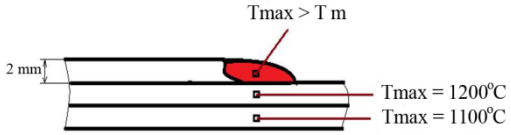

Figure 3. Temperature of the previous rollers under the weld pool

Thus, if the nitrogen content is [N] ≥ 0.03 wt. % in the initial filler material, or oxygen in [O] ≥ 0.02 wt. % in the welded metal of heat-resistant nickel alloys at a temperature of 1100°C, a significant decrease in its deformation capacity (ε ≤ 3.8%) is recorded.

Conclusions

1. It has been established that the content of active gases ([O] and [N]) in the powder of the ЭП 648 ВИ alloy significantly affects the mechanical properties of the workpieces obtained by the plasma additive growth method. At the same time, the chemical composition of the deposited metal is determined by the chemical composition of the powder, which is confirmed by minimal deviations within 1-5%.

2. It has been proved that an excess of nitrogen [N] ≥ 0,03 wt.% or oxygen [O] ≥ 0,02 wt.% in the powder of the ЭП-648ВИ alloy leads to the formation of hot cracks in the deposited metal at a temperature of 1100°C. This causes a significant decrease in its deformation capacity (ε ≤ 3,8%), which, in turn, affects the reduction of strength and ductility in the longitudinal direction.

3. The microstructure of samples with a high content of active gases shows a tendency to form cracks that develop along grain boundaries. In the cross direction, the samples retain better mechanical properties due to less cracking. The cracks have melted edges characteristic of hot cracks, which confirms the fracture mechanism of «ductility dip cracking».

4. The content of oxygen and nitrogen in the deposited metal increases by 20-30% compared to the powder, which indicates the importance of argon quality and stability of its supply. The use of high-quality shielding gas is critical to reducing the risk of defects.

The results of the study can be used to improve the technology of plasma powder surfacing by:

- selection of powders with acceptable limits of active gas content ([O] ≤ 0,02 wt.%, [N] ≤ 0,03 wt.%);
- ensuring a stable thermal cycle during surfacing to avoid hot cracks;
- use of high-quality protective argon to minimise the penetration of oxygen and nitrogen into the weld metal.

Thus, the results of the study provide a scientific and technical basis for optimising the conditions of additive growth, which allows to improve the quality and reliability of aircraft parts.

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ОЦІНКА ВПЛИВУ ВМІСТУ АКТИВНИХ ГАЗІВ У ПОРОШКУ НА МЕХАНІЧНІ ВЛАСТИВОСТІ ЗАГОТОВОК ДЕТАЛЕЙ, ОТРИМАНИХ МЕТОДОМ ПЛАЗМОВИХ АДИТИВНИХ ТЕХНОЛОГІЙ

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***Мета роботи**. Аналіз впливу якості порошку (вміст активних газів) на рівень механічних властивостей заготовок, отриманих методом плазмових адитивних технологій.*

***Методи дослідження**. Хімічний склад порошків визначали у відповідності за нормативними документами: ГОСТ 17745, ОСТ 1 90134, ОСТ 1 90136, ОСТ 1 90137, ОСТ 1 90138.*

Наявність неметалевих включень і забруднень в порошках визначали шляхом зовнішнього огляду.

Для кількісної оцінки кисню у порошках використовували метод вакуумної екстракції у відповідності із ДСТУ ISO 4491-4:2016. Газоаналізатор кисню і азоту ТС 500 (фірма LECO). Перед завантаженням у дозатор установки усі порошки пройшли операцію просушування у печі (температура – 250°С, впродовж 1 години).

Зразки (розмір 130×70×14 мм) були виготовлені адитивним вирощуванням методом плазмової порошкової наплавки (МПН) на спеціалізованій роботизованій установці STARWELD 190H.

Хімічний склад вирощених зразків визначали спектральним методом з використанням оптико-емісійного спектрометру SPECTROMAX за стандартними методиками у відповідності з ГОСТ19863.1-19863.12.

Вміст азоту та кисню у наплавленому металі визначали у відповідності ТС 500 (фирма LECO).

Мікроструктурний аналіз проводився на шліфах у повздовжньому та поперечному напрямах до та після термообробки, після травлення у реактиві для електротравлення жароміцних сплавів (H_3PO_4 – 800мл+CrO_3 – 100мл).

Механічні властивості сплаву визначали на стандартних циліндричних зразках по ГОСТ 1497-84, ГОСТ 1497-84, ГОСТ 10145-81.

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Отримані результати. *Хімічний склад наплавленого металу задається хімічним складом порошку. Вміст кисню та азоту у наплавленому металі задається вмістом кисню та азоту у самому порошку, а також захистом при наплавленні зварювальної ванни аргоном. Встановлено, що в сплаві ЭП-648ВИ при вмісті у порошку з азоту у кількості [N] ≥ 0,03 ваг. %, або кисню у [O] ≥ 0,02 ваг. % в наплавленому металі при температурі 1100°С фіксується значне зниження його деформаційної здатності (ε ≤ 3,8 %), що під вливом термічних напруг призводить до появи гарячих тріщин, а як наслідок у поздовжньому напрямі ми бачимо значне падіння міцності та пластичності.*

Наукова новизна. *Встановлено вплив якості порошку сплаву ЕП-648ВИ на властивості заготовок, отриманих за адитивними технологіями.*

Практична цінність. *Встановлений в роботі негативний вплив вмісту активних газів на механічні властивості вирощених заготовок дозволить на стадії вхідного контролю відсіяти порошок з неприпустимім вмістом активних газів ([N] ≥ 0,03 ваг. %, або кисню у [O] ≥ 0,02 ваг. %).*

Ключові слова: *адитивні технології, плазмове наплавлення, механічні властивості, авіаційні двигуни, вирощування.*

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